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ADL-4D6: A SILICA/SILICA COMPOSITE FOR HARDENED ANTENNA WINDOWS--ETC(U)
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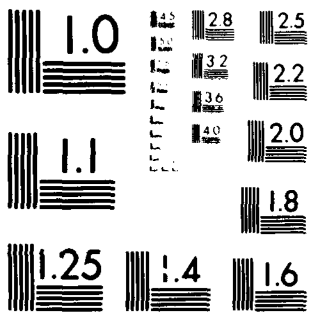

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SUMMARY

A silica/silica composite designated "ADL-4D6" has been developed at GE/RESD for advanced missile shock-resistant antenna window applications.* It is based on RESD's "Omniweave" 4-D reinforcement system using high purity fused silica fibers, densified to 1.6 gm/cc by repeated infiltration with an aqueous suspension of colloidal silica. This paper describes an improved fabrication process, from both the performance and cost standpoint, and summarizes mechanical, thermal and electrical design data.

The results of flyer plate testing show a spall threshold of 7300-8000 taps for a 1 inch thickness, the highest spall resistance ("hardening") of any known inorganic dielectric material. Minimum flexure strengths of 5000 psi and 1.0% strain to failure have been achieved. ADL-4D6 can thus be considered a "ductile ceramic".

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KEY WORDS

Silica, dielectric, composites, microwave materials

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ADL-4D6: A Silica/Silica Composite for Hardened Antenna Windows

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Introduction

This paper describes the development of a new inorganic silica/silica composite for hardened antenna window (HAW) and radome applications. ADL-4D6 is one of a series of such hardened, or mechanical shock resistant radar-transmitting materials developed at GE/RESD under contract to the U.S. Army Materials and Mechanics Research Center (AMMRC) since 1969. The development of a previous silica fiber reinforcement/silicone resin matrix composite designated ADL-10 has been reported in References 1 and 2.

The primary Army application for such materials is for antiballistic missiles (ABM); the materials are however also quite suitable for ballistic re-entry vehicle and tactical missile applications. The performance requirements for HAW's are summarized in Table 1.

Table 1
Materials Performance Goals for Hardened Antenna Windows

Performance Category	Related Materials Property Goal
RF Transmission during Hypersonic Aerodynamic Heating	Loss Tangent $<$ 0.01 maintained
Hardening - Shock Impact Resistance	Order of magnitude improvement over conventional materials (e.g., fused silica spalls at 500-1500 taps in flyer plate tests)
Structural/Mechanical	Strength and modulus comparable to conventional materials, but order of magnitude improved strain to failure - "fracture toughness", (fused silica failure strain = 0.01-0.07%)
Ablation/Thermal	Equivalence with conventional materials of same chemical composition
Environmental	Free from humidity and aging effects, (especially the electrical properties, above)

The obvious method of obtaining such improved mechanical and shock resistance properties has been to develop multidirectional fiber-reinforced composites based on the conventional dielectric materials, such as silica. The "obvious" aspect of this approach was the choice of the all inorganic silica/silica composite approach; the realization of such an inorganic composite material has been far more arduous. Our efforts to develop "Markite", an all inorganic silica/silica based on repetitive pyrolysis and reimpregnation of the ADL-10 material, have been described in references 1, 3 and 4. All of our approaches at GE/RESD have been based on the use of "Omniweave" woven preforms or reinforcements of J.P. Stevens "Astro-quartz" high purity fused silica. Figure 1

shows the 4-D reinforcement geometry and a photo of a silica Omniweave. In the Markite material, the silica matrix was found to bind too tightly to the silica fiber system, causing fiber damage (see Figure 6 of Ref. 1). Also, the dense, rigid matrix had too high an effective shear modulus, so that the desired composite fiber/matrix shear interaction did not occur in this material.

ADL-4D6: Process Development

The matrix of the present material, ADL-4D6, is adapted from the aqueous colloidal silica densification process previously developed by Philco-Ford Aeronutronics under contract to the USAF (Ref. 5). AS-3DX is made by densifying a 3-D silica preform by repetitive cycles of infiltration with Ludox aqueous silica colloidal suspensions, and drying and sintering to a rigidized body. The ADL-4D6 composite is based on 4-D silica Omniweave densified with this colloidal silica process, with the additional modifications indicated in Figure 2. Starting with reinforcement fabric densities of from 1.0 to 1.2 gm/cc, final densities of from 1.5 to 1.6 are attained, in five to seven cycles. A photograph of a densified ADL-4D6 sample surface is shown in Figure 3.

The current "optimized" process includes some significant improvements which can be identified at stages in the flow chart of Figure 2. First, an extra low sodium "AS" version of the Ludox sol has been made available by DuPont, and ADL-4D6 made from it has shown equivalent mechanical and electrical performance in a screening test program, as compared to material produced via a rather long process of ion exchange purification of the lowest sodium content Ludox previously available. We have found, then, that this version of Ludox AS can be used directly as received, for a significant cost and schedule savings.

Second, the application of a silane "coupling agent" to the silica preform before the first Ludox densification cycle has been found to yield further improvement in mechanical strengths. We also have some indications of achieving higher strains to failure because of this process modification, but the experimental difficulties of accurately measuring strains already in excess of 1% have prevented a conclusive demonstration.

A third modification is a "water desensitization process" for the densified composite. The highly active, porous and permeable sintered matrix silica of this material is known (ref. 5) to have a high affinity for adsorbed atmospheric water vapor, which reacts with the highly polar surface to form stable hydroxyl ions, resulting in an unacceptably high electrical loss. To minimize this effect, techniques were developed to react the adsorbed surface water layer with a halogenated silane vapor to form a polysiloxane coating in place of the water. The applied quantity of this reactant was minimized to reduce the amount of any residual free carbon that would be formed during high temperature pyrolysis.

ADL-4D6 Properties

RF dielectric constant and loss tangent measurements made by a new laser irradiation technique simulating hypersonic flight heating have shown retention of a loss tangent less than 0.01 at temperatures above fusion (Table 2). This technique, in which the specimen is irradiated by the CW laser in a waveguide, so that accurate simultaneous transmission and reflectance measurements can be made, is described in Ref. 6 along with more detailed microwave property data.

A set of typical uniaxial stress-strain curves is shown in Figure 4. Note the non-linear deflection and pseudo-plasticity evident. A set of MOR (modulus of rupture) values for the material was generated in an extensive characterization program over temperatures from -100 to 1550°F, in three plate orientations.

These mechanical data are summarized in Table 2 along with other design property data for ADL-4D6. The axial strength MOR data are presented in Figure 5.

A fracture surface of a typical specimen is shown in the SEM (scanning electron microscope) photograph of Figure 6. The SEM shows an ideal composite fracture surface with individual filaments pulled out from the matrix, in contrast to the smooth, brittle fracture surfaces one would find in a monolithic fused silica or our earlier unsuccessful Markite composite silica. The set of specimens from which this SEM was made yielded room temperature flexure test MOR values in the range 10,000-40,000 psi. These near theoretical strengths cannot be consistently reproduced but the current version of the material shows minimum MOR values in excess of 5,000 psi. ADL-4D6 has a minimum strain capability of 1.0% in this plate axial direction, a remarkable property for an inorganic composite of silica-literally a "ductile ceramic".

Consistent with the intended application as a HAW, an extensive characterization of the mechanical shock and spall resistance of ADL-4D6 has also been conducted (Ref. 6). Exploding foil flyer impact tests show a spall threshold in excess of 7.3 kilotaps for a one inch thickness of material judging by a front face crushing criterion; and above 8 kilotaps using a backface spall criterion. This is an order of magnitude better than the spall resistance that conventional monolithic fused silicas offer and is attributable to the combination of high tensile strength and attenuation of shock waves in the composite.

A small number of particle impact tests have also been conducted and a more extensive program of this type is planned for future development.

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Table 2

Properties of ADL-4D6 Silica/Silica Composite Antenna Window Material

Property	-100	RT	700	1300	1500
		Temperature, ^{o}F			
Ultimate Tensile Strength* (psi)					
-- plate axial direction**	8000	5000	7000	10,000	9,000
-- plate body diagonals		5000	6000		
Elastic Modulus, Axial, 10^6 psi	2.3	1.1			
Axial Tensile Failure Strain (%)	1.0	1.1			
Compressive Strength, Axial*	10,000	10,000	12,000	12,000	14,000
Compressive Modulus, Axial*	1.2	1.2			
Torsional Shear Strength/Modulus/ Failure Strain, At RT		3650/0.73 x 10^6/0.55%			
Thermal Expansion Coefficient (mean, 70 - 1300^{o}F, Axial)		0.28 x 10^{-6} / F^o			
Thermal Conductivity plate axial direction, BTU/ft. hr. F^o	0.32	0.39	0.46	0.54	0.58
Dielectric Constant (range)		2.8 - 3.1			
Loss Tangent (max.) (at 250 MHZ)		0.006 (less than 0.01 through melting temperature)			
Density, Bulk, gm/cc		1.6			

* Strength and strain data are minimum allowables, elastic moduli are mean values.

** "Plate axial directions": along width, length and thickness directions of a densified rectangular plate; the principal fiber reinforcement directions of the 4-D system would then be in the 4 "plate body diagonal" directions.

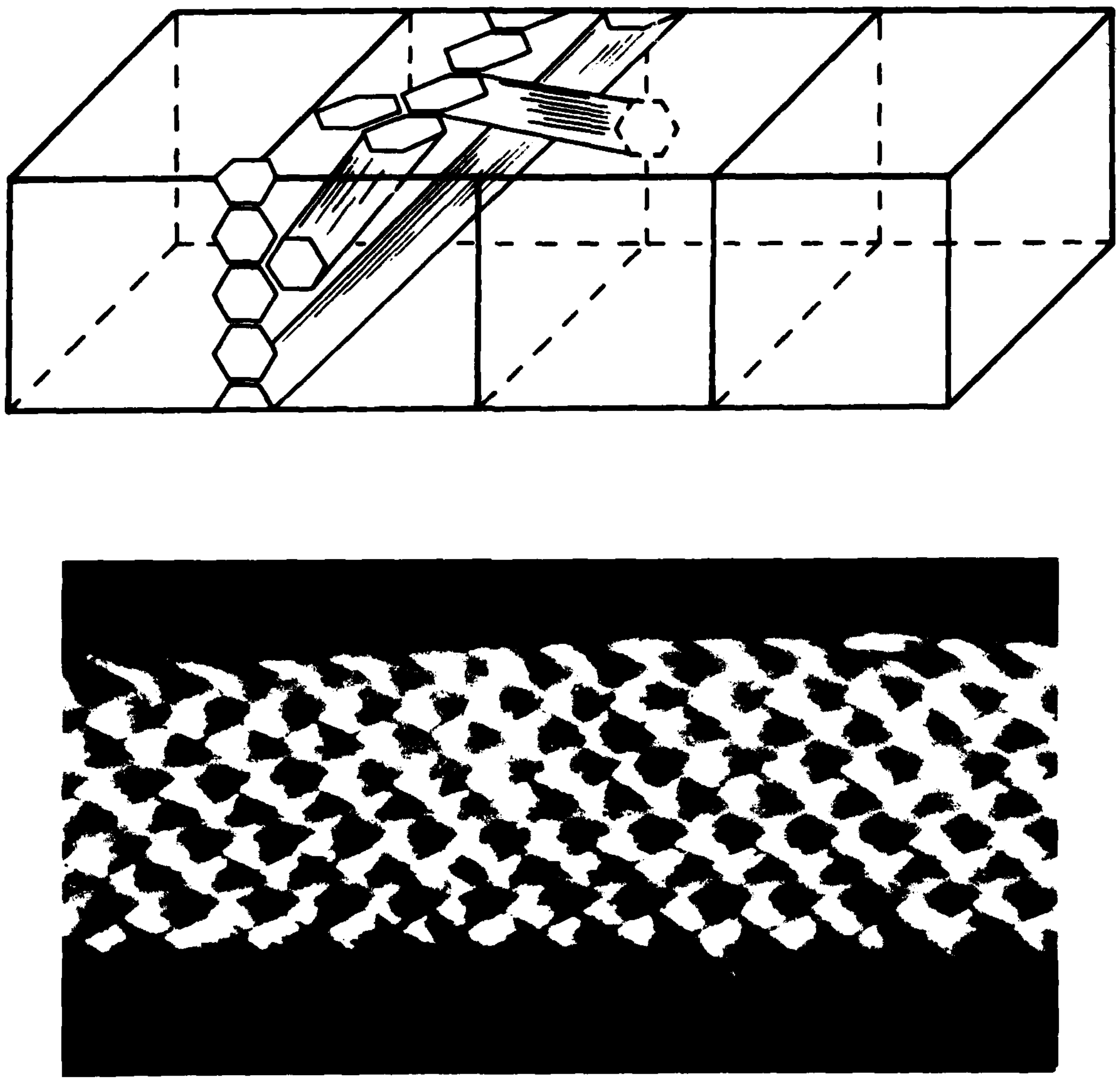

FIGURE 1. ISOMETRIC VIEW AND PHOTO OF 4–D CUBIC SILICA OMNIWEAVE.

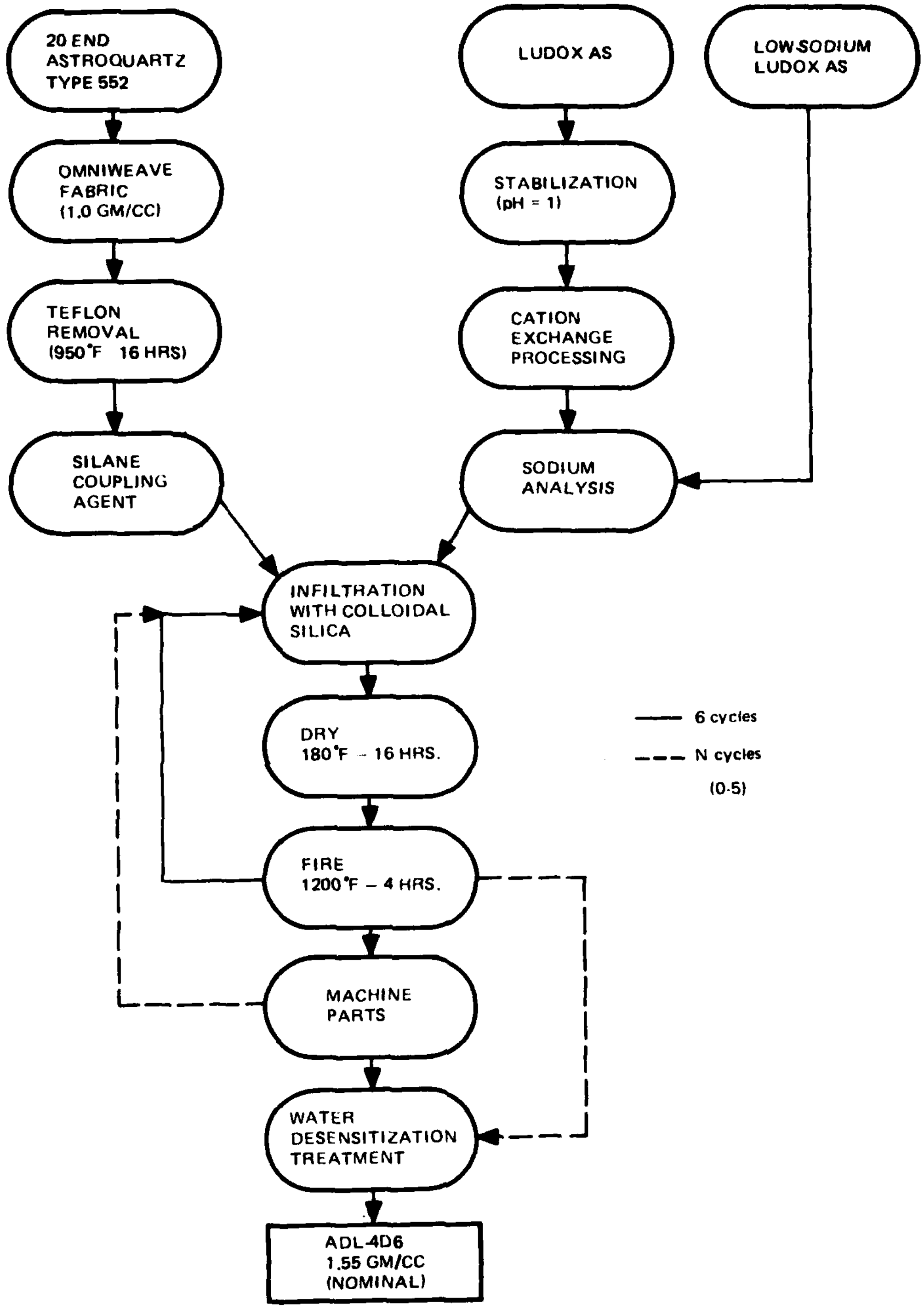

Figure 2. ADL-4D6 Fabrication Process (B).

Figure 3. Photomicrograph of Polished Section of ADL–4D6, 0.5 to 0.4

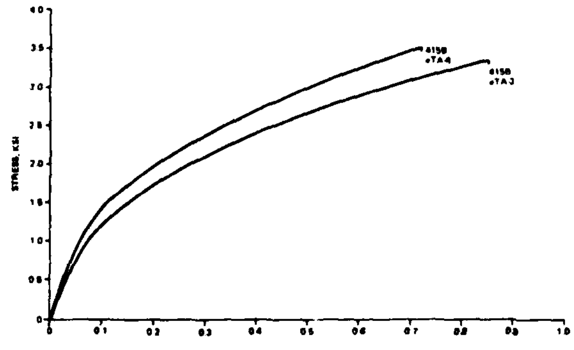

Figure 4. ADL–4D6 Typical Stress-Strain Curves for Rectangular Section Specimens (0.40" thick).

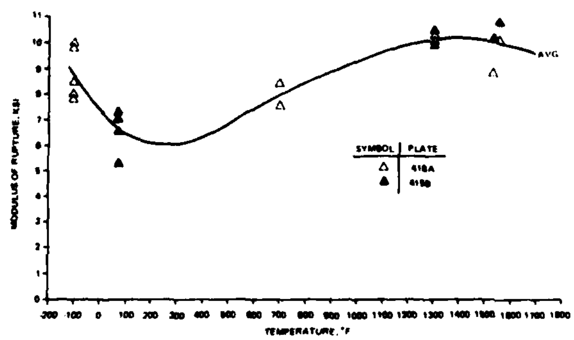

Figure 5. ADL–4D6 Design Axial Flexure Data.

FIGURE 6. SEM PHOTO OF ADL—4D6 SILICA/SILICA COMPOSITE.